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Abstract: This study evaluated the effect of air-abrasion protocols on the topography, surface wettability and adhesion of resin cement to zirconia. Ceramic specimens (N = 49; n = 7) (15 mm × 2 mm) were randomly allocated to seven groups to be treated with: (1) Air-abrasion with 45 m Al2O3 (A45), (2) 80 m Al2O3 (A80), (3) 30 m Al2O3 coated with SiO2 (CoJet) (C30), (4) 30 m Al2O3 coated with SiO2 (Rocatec Soft) (R30), (5) 110 m Al2O3 coated with SiO2 (Rocatec Plus) (R110); (6) R110R30 (Rocatec) (R110R30) and (7) control, no conditioning (NC). Air-abrasion was performed using a chairside air-abrasion device (2.5 bar, 10 mm, 90 s). Contact angle measurements were performed using goniometry (n = 5). MDP-based dual resin cement (Panavia F2.0) was bonded on four locations after air-abrasion protocols (n = 20 per group). Half of the specimens were tested after 24 h and the other half after thermal cycling (×3000, 5–55 °C). Data were analyzed using 1-, 2-way ANOVA and Tukey's test (alpha = 0.05). Significantly lower contact angle values were observed for groups C30 (62.6 ± 0.91), R30 (61.91 ± 1.05) and R110R30 (61.54 ± 1.02) compared to those of other groups (65.5 ± 0.9–110.61 ± 0.9) (p < 0.05). In dry conditions, surface conditioning methods tested did not show significant effect on bond strength (MPa) (10.57 ± 1.42–16.86 ± 2.54) (p = 0.238). After thermocycling, bond strength results decreased significantly (p < 0.05) (12.6–51.2%). R110 (7.18 ± 1.34) and A80 (4.92 ± 1.53) showed significantly higher bond strength compared to other groups (2.13 ± 0.73–4.16 ± 1.34) (p < 0.05). The best wettability and adhesion results with MDP-based resin cement to zirconia was achieved with A80 and R110 air-abrasion.

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Effect of different air-abrasion protocols on topography, surface wettability and adhesion of MDP monomer-based resin cement to zirconia

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Short title: *Air-abrasion protocols for yttrium stabilized zirconia*

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Abstract: This study evaluated the effect of air-abrasion protocols on the topography, surface wettability and adhesion of resin cement to zirconia. Ceramic specimens (N=49; n=7) (15 mm x 2 mm) were randomly allocated to 7 groups to be treated with: 1) Air-abrasion with 45 μm Al_2O_3 (A45), 2) 80 μm Al_2O_3 (A80), 3) 30 μm Al_2O_3 coated with SiO_2 (CoJet) (C30), 4) 30 μm Al_2O_3 coated with SiO_2 (Rocatec Soft) (R30), 5) 110 μm Al_2O_3 coated with SiO_2 (Rocatec Plus) (R110); 6) R110R30 (Rocatec) (R110R30) and 7) control, no conditioning (NC). Air-abrasion was performed using a chairside air-abrasion device (2.5 bar,10 mm, 90 s). Contact angle measurements were performed using goniometry (n=5). MDP-based dual resin cement (Panavia F2.0) was bonded on four locations after air-abrasion protocols (n=20 per group). Half of the specimens were tested after 24 h and the other half after thermal cycling (x3000, 5-55°C). Data were analyzed using 1-, 2-way ANOVA and Tukey's test (alpha=0.05). Significantly lower contact angle values were observed for groups C30 (62.6±0.91), R30 (61.91±1.05) and R110R30 (61.54±1.02) compared to those of other groups (65.5±0.9 - 110.61±0.9) ($p<0.05$). In dry conditions, surface conditioning methods tested did not show significant effect on bond strength (MPa) (10.57±1.42 - 16.86±2.54) ($p=0.238$). After thermocycling, bond strength results decreased significantly ($p<0.05$) (12.6-51.2%). R110 (7.18±1.34) and A80 (4.92±1.53) showed significantly higher bond strength compared to other groups (2.13±0.73 - 4.16±1.34) ($p<0.05$). The best wettability and adhesion results with MDP-based resin cement to zirconia was achieved with A80 and R110 air-abrasion.

Keywords: Adhesion; Air-abrasion; Resin cements; Surface conditioning; Zirconium dioxide

Introduction

Among ceramic materials, partially stabilized by yttria stabilized zirconia ceramic (Y-TZP; hereon: zirconia) undergo transformation change that is a property that allows for increased mechanical strength making the material suitable for the indications in load-bearing areas in reconstructive dentistry [1-4].

Since f zirconia is composed of crystalline phase without silica, surface conditioning with 5-10% hydrofluoric acid is not effective for surface roughening [5]. For this reason, various surface conditioning methods have been proposed in order to improve the adhesion of resin cements to zirconia, among which are airborne particle abrasion with aluminium oxide (Al_2O_3), Al_2O_3 coated with silica (Si_2O_3), application of specific primers or glazing the intaglio surface of zirconia reconstructions [5,6]. Current knowledge dictates that micromechanical retention through air-abrasion is still the most effective method for conditioning zirconia which could be accomplished either chairside or at the dental laboratory using particles at different sizes [7].

The effect of air-abrasion procedures on zirconia ceramic surface depends on the particle type, size, pressure and the distance of the nozzle to the surface [8,9]. Al_2O_3 particles of 50 to 250 μm size are commonly used as they can be obtained at low costs globally. However, Al_2O_3 particles were reported to generate microcracks in ceramics [10]. The type and size of the particles used in conjunction with air-abrasion methods may affect the bond strength values where larger particles promote cracks in the material which may lead to failures at the adhesive interface [11,12]. On the contrary, smaller particles and those that are coated with silica may increase the adhesion of resin cements based on 10-Methacryloyloxydecyl dihydrogen phosphate monomer (MDP), with more hydrolytic stability [13]. Due to the impact during air-abrasion, silica layer on the alumina particles remain attached on the surface [12], which then reacts with the adhesive promoter or resin cement, a process called silicatization [13]. While laboratory air-abrasion system is based on utilizing 110 μm alumina particles to clean and roughen the surface followed by the application of 30 μm silica particles (Rocatec, 3M ESPE), the chairside application of the same method is based on deposition of 30 μm silica particles only (CoJet, 3M ESPE). The particle size and morphology may impair mechanical stability of zirconia where larger particles were reported to cause more monoclinical phase transformation [11,12]. Recently, similar air-abrasion methods are employed for roughening zirconia implant surfaces and there is limited information available on the best deposition method and particle size to achieve increased wettability which is crucial for the osseointegration.

The objectives of this study therefore were to study the effect of air-abrasion protocols on the topography, surface wettability, adhesion of MDP-based resin cement to zirconia and evaluate failure types after debonding. The null hypothesis tested was that particle size would not significantly affect the surface wettability and adhesion of resin cement tested to zirconia.

Materials and Methods

Specimen preparation

Zirconia specimens (N=49) were fabricated according to the manufacturer's recommendations (LAVA All-Ceramic System, 3M ESPE, St. Paul, USA) (diameter: 15 mm; thickness: 2 mm). The specimen surfaces were finished using silicon carbide papers under water-cooling in the order of 800, 1200, 1500, 2000-grit prior to sintering. The specimens were randomly divided into 7 groups depending on the surface conditioning methods:

Surface conditioning methods

Air-abrasion was carried out using a chairside device (Microjato Standard, Bioart, Sao Paolo, Brasil) [14] operating at 2.5 bar from a distance of 10 mm in circular motions for 90 s with the following particles: 1) 45 μm Al_2O_3 (A45) (Polidental, São Paulo, Brasil), 2) 80 μm Al_2O_3 (A80) (Polidental), 3) 30 μm Al_2O_3 coated with SiO_2 (CoJet, 3M ESPE) (C30), 4) 30 μm Al_2O_3 coated with SiO_2 (Rocatec Soft) (R30) (3M ESPE), 5) 110 μm Al_2O_3 coated with SiO_2 (Rocatec Plus, 3M ESPE) (R110, 6) R110+R30 (Rocatec System, 3M ESPE) (R110R30), and 7) control, no conditioning (NC).

Surface wettability measurements

Two specimens from each group were selected and contact angle measurements were performed using a goniometer (Contact Angle Goniometer, Rame-Hart Inc., Montain Lakes, USA) at controlled temperature and humidity. Measurements were made using the corresponding software (RHI 2001 Software Imaging, Montain

Lakes, USA). A drop of deionized water was applied on the ceramic surface using a syringe and the contact angle was measured for 20 s (30 frames per second) after the initial break of 10 s.

Bonding procedures

After surface conditioning, a tape with three holes were placed (diameter: 3 mm) (Scotch Tape, 3M ESPE, Sumare, SP, Brazil) on the zirconia specimens. Then, silane coupling agent was applied one coat (Monobond S, Ivoclar Vivadent, Schaan, Liechtenstein) with a microbrush (Vigodent, Rio de Janeiro, RJ, Brazil), waited for its reaction for 60 s and then air-dried for 20 s. On each hole a cylindrical transparent plastic mould (Saint-Gobain Performance Plastic, Maime Lakes, FL, USA) was fixed (inner diameter: 3 mm; height: 3 mm) with cyanoacrylate gel (Super Glue, Loctite, Diadema, SP, Brazil). MDP containing resin cement (Panavia F2.0, Kuraray, Okayama, Japan) was applied into the mould with the aid of a centrix syringe (AccuDose needle, Polidental Ind. E Com. Ltd., São Paulo, Brazil). Resin cement was photo-polymerized using an LED polymerization device (Tygon Radii-Cal LED, SDI Pines, SP, Brazil) at an intensity of 1200 mW/cm^2 for 40 s from each side. After removal of moulds, half of the specimens were tested after 24 h and the other half after thermal cycling (x3000, 5-55°C; dwelling time in each bath: 30 s) (Ética Equipamento Científicos S/A, São Paulo, Brasil).

Macroshear tests

The specimens were loaded under shear at the ceramic-resin interface in a Universal Testing Machine (EMIC DL-2000, Company, Sao Paolo, Brasil) using a wire (diameter: 0.4 mm) around the cement, making contact with the substrate. The shear force (20 Kgf) was applied at a cross-head speed of 0.5 mm/min until failure.

Microscopic examination and failure analysis

After each air-abrasion protocol, images were made (n=2 per group) from 2 randomly selected specimens using Scanning Electron Microscope (SEM) (JSM-5600 LV, Jeol, Tokyo, Japan) in order to observe topographical changes [15].

After adhesion tests, the debonded specimen surfaces were examined in order to analyze the failure types using an optical microscope (Zeiss MC 80 DX, Jena, Germany) at x50 magnification. Failure types were planned to be classified as follows: Score 1: Adhesive failure at the ceramic-cement interface with no cement remnants left on the substrate, Score 2: Cohesive failure within the cement, Score 3: Cohesive failure within the substrate, Score 4:<1/3 cement left adhered on the substrate.

Statistical analysis

Statistical analysis was performed using Statistica 8.0 software for Windows (StatSoft, Inc., Tulsa, OK, USA). Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data. As the data were normally distributed, 2-way ANOVA and Tukey`s tests were used where the bond strength was the dependent variable and conditioning methods (7 levels: A45, A80, C30, R30, R110, R110R30, NC), and aging types (2 levels: dry versus thermocycle) as independent variables. In addition, 1-way ANOVA and Tukey`s tests were used for the statistical analysis of the wettability data. P values less than 0.05 were considered to be statistically significant in all tests.

Results

Air-abrasion type influenced the contact angle values and bond strength results significantly ($p<0.05$). Interaction terms were also significant ($p<0.05$).

Significantly lower contact angle values were observed for groups C30 (62.6±0.91), R30 (61.91±1.05) and R110R30 (61.54±1.02) compared to those of other groups (65.5 ±0.9 - 110.61±0.9) ($p<0.05$) (Table 1). Mean contact angle value was significantly higher in the control group (110.61±0.9) ($p<0.05$).

In dry conditions, surface conditioning methods tested did not show significant effect on bond strength (10.57±1.42 - 16.86±2.54 MPa) ($p=0.238$) (Table 2). After thermocyling, bond strength results decreased significantly in all groups ($p<0.05$) ranging between 12.6 to 51.2% (Fig. 1).

Among all air-abraded groups, R110 (7.18±1.34) and A80 (4.92±1.53) showed significantly higher bond strength values compared to other groups (2.13±0.73 - 4.16±1.34 MPa) ($p<0.05$).

Failure types were exclusively adhesive in all groups.

SEM images (x2000) showed evident traces of silicon carbide paper in the control group and in A45, A80, C30, R30 specimens while the increase in particle size (R110 and R110R30) created more number of grooves and irregularities (Figs. 2a-g). No cracklines were observed in any of the specimens.

Discussion

This study evaluated the effect of air-abrasion protocols on the topography, surface wettability, adhesion of MDP-based resin cement to zirconia. Based on the results of this study, since air-abrasion method significantly affected the surface wettability and adhesion of resin cement tested, the null hypothesis could be rejected.

Reliable adhesion between the resin cement and zirconia is a prerequisite especially for the clinical longevity of minimal invasive reconstructions [15,16]. Studies have focused on the selection of cement types in order to obtain adequate adhesion to intaglio surfaces of zirconia frameworks [15,17,18] but the most appropriate cementation protocol is not yet consolidated [19]. Due to its composition, microstructure and physical properties, durable adhesionwith conventional cements could not be obtained and therefore zirconia surfaces need to be conditioned prior to cementation [7,20-22]. Among the proposed surface conditioning methods, especially air-abrasion protocols have been reported to increase the surface area and surface energy and promote mechanical microretentions that allow for cement interlocking [9,22-24]. Morphological changes through air-abrasion also alter wettability of the substrate surface [25]. These findings are confirmed in this study where higher wettability and adhesion was obtained after air-abrasion protocols when compared to the non-conditioned control group.

In the literature, while some studies reported favourable results with the use of aluminum oxide particles [8,23,26,27], others suggested that such particles may damage zirconia creating microcracks and thereby reducing the mechanical resistance of restorations by 20 to 30% [10,11,28,29]. These aspects are directly related to the size and type of particles employed during air-abrasion. According to Kosmac et al., aluminum oxide could remove a layer of 60 µm from the ceramic surface [30]. This superficial damage, although microscopic, negatively influences the mechanical stability of zirconia [31,32]. Moreover, excessively high pressure practiced during air-abrasion may initiate phase transformation, and accelerate crack formation [10]. Likewise, particle size and deposition duration may also affect the stability of zirconia [10,33-35]. Given this fact, in this study particle deposition was achieved under the same pressure, nozzle distance and duration while particle size varied.

In fact, high-strength ceramics such as zirconia are hard materials and therefore they cannot be effectively air-abraded [36,37]. For this reason, abrasion with larger particles produce more roughness, and hence better micromechanical retention. According to results obtained after thermocycling, A80 and R110 groups delivered statistically similar bond strength values, being higher than those of other groups. Similarly, Özcan et al. found no statistical differences between Al_2O_3 and Al_2O_3 coated with SiO_2 in dry conditions but after thermocycling the latter showed less hydrolytic degradation [19]. In this study, A80 containing less aggressive particles compared to 110 μm helped for better wettability and therefore less decrease in bond strength.

The increase in surface energy of zirconia after mechanical and chemical conditioning may improve the union between the resin-based cements and zirconia [38]. The topographic features observed in A80 and R110 groups with larger particles produce greater irregularities, and hence higher surface roughness [14],. It should be noted that local changes in the surface energy of solid substrates may affect contact angle values. In this context, wettability properties change as a function of contact angle hysteresis. Hence, rough surfaces tend to show high hysteresis of contact angle by air entrapment in the deeper parts of the valleys created by the particles. This fact may explain the differences in values observed for A45 groups, R30 and C30 where more homogeneous surfaces were observed.

R110R30 particle type on the other hand, combines the advantages of larger and smaller particles. However, contact angle, bond strength values and topographical changes observed with this particle type were similar to groups with smaller particles namely, R30 and C30. While air-abrasion with Al_2O_3 coated SiO_2 provides ultrafine mechanical retention and welding of silica particles [17], these particles may detach from the surface over time [13,22,27,39]. One other factor is the hydrolytic degradation of zirconia-resin cement interface [27,40,41]. This could explain the reasons for the loss of bond strength in this study after aging which supports previous findings

[13,42,43]. In this study, thermocycling was limited to 3000 cycles based on pilot studies but prolonged number of cycles may further decrease the achieved results.

As for the analysis of failures, all groups presented adhesive failures. The absence of cohesive or mixed failures [16,44-46] demonstrates that the shear stress applied to the zirconia-resin interface could detach the cement from the zirconia surface completely which is an indication of insufficient adhesion to zirconia.

Based on the results of this study, it is worth noting that the different particle size and morphology directly affected the wettability and bond strength results. Although particle deposition with smaller particles resulted in more homogeneous surface topography and increased wettability, bond strength results after aging were higher for the groups conditioned with larger particles. To the authors\` best knowledge, this is the first study that investigated all types of sand particles ranging from small to large size available for laboratory and chairside applications in one study. Especially the use of 80 μm Al_2O_3 (A80) has not been studied in earlier studies. Since silica coated alumina particles are not available universally, based on non-significant results, the use of A80 could substitute CoJet or Rocatec systems. Clinical studies should verify whether the achieved bond strength results are sufficient for longevity of minimal invasive zirconia reconstructions and the wettability results for biological interactions with zirconia implants.

Conclusions

From this study, the following could be concluded:

1- The non-conditioned control group resulted in the least wettability and delivered the lowest bond strength values of the tested MDP-based resin cement to zirconia.

2- In non-aged conditions, all types of air-abrasion particles tested showed similar bond strength results.

3- Aging decreased the bond strength results after all air-abraded groups with the A80 and R110 showing the highest results.

Clinical Relevance

Air-abrasion of zirconia surfaces increased the wettability and adhesion of 10-MDP based resin cement. Aging decreased the achieved bond strength where 80 µm Al_2O_3 and 110 µm Al_2O_3 coated with SiO_2 (Rocatec Plus) showed the highest bond strength. Yet, failure types were adhesive in all groups, indicating weak adhesion to zirconia.

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Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

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Captions to figures and tables:

Tables:

Table 1. Mean±standard deviations of the contact angle (°) measurements. Different superscript letters in one column represent statistical significance ($p<0.05$). A45: Air-abrasion with 45 µm Al_2O_3, A80: 80 µm Al_2O_3, C30: 30 µm Al_2O_3 coated with SiO_2 (CoJet), R30: 30 µm Al_2O_3 coated with SiO_2 (Rocatec Soft), R110: 110 µm Al_2O_3 coated with SiO_2 (Rocatec Plus), R110R30: R110+R30 (Rocatec System), NC: Control no conditioning.

Table 2. Mean macroshear bond strength values (MPa±standard deviations) without and with thermocycling. *Uppercase letters in one column represent statistical significant differences and lower case letters in one row ($p<0.05$). See Table 1 for group abbreviations.

Figures:

Fig. 1 Bond strength change in percentage between non-aged and aged groups.

Figs. 2a-g SEM images (x2000) of **a)** NC, **b)** A45, **c)** A80, **d)** C30, **e)** R30, **f)** R110, **g)** R110R30. Note the evident traces of silicon carbide paper in the control group and in A45, A80, C30, R30 specimens while the increase in particle size with R110 and R110R30 created more roughness and surface irregularities. See Table 1 for group abbreviations.

Tables:

Experimental Groups	Contact Angle (°)
	Mean±Standard Deviation
NC	110.61±0.9[a]
A45	65.50±0.94[d]
A80	78.37±0.62[c]
C30	62.66±0.91[de]
R30	61.91±1.05[e]
R110	80.68±0.84[b]
R110R30	61.54±1.02[e]

Table 1. Mean±standard deviations of the contact angle (°) measurements. Different superscript letters in one column represent statistical significance ($p<0.05$). A45: Air-abrasion with 45 μm Al_2O_3, A80: 80 μm Al_2O_3, C30: 30 μm Al_2O_3 coated with SiO_2 (CoJet), R30: 30 μm Al_2O_3 coated with SiO_2 (Rocatec Soft), R110: 110 μm Al_2O_3 coated with SiO_2 (Rocatec Plus), R110R30: R110+R30 (Rocatec System), NC: Control no conditioning.

Experimental Groups	Dry	Thermocycling
	Mean±Standard Deviation	**Mean±Standard Deviation**
NC	10.5 ±1.42[Aa]	1.86±0.30[Cb]
A45	14.38±2.96[Aa]	3.75±1.94[Bb]
A80	12.41±2.08[Aa]	4.92±1.53[Ab]
C30	16.86±2.54[Aa]	2.13±0.73[Cb]
R30	13.98±3.55[Aa]	4.16±1.34[Bb]
R110	14.01±2.03[Aa]	7.17±1.34[Ab]
R110R30	11.74±2.2[Aa]	3.25±1.37[BCb]

Table 2. Mean macroshear bond strength values (MPa±standard deviations) without and with thermocycling. *Uppercase letters in one column represent statistical significant differences and lower case letters in one row ($p<0.05$). See Table 1 for group abbreviations.

Figures:

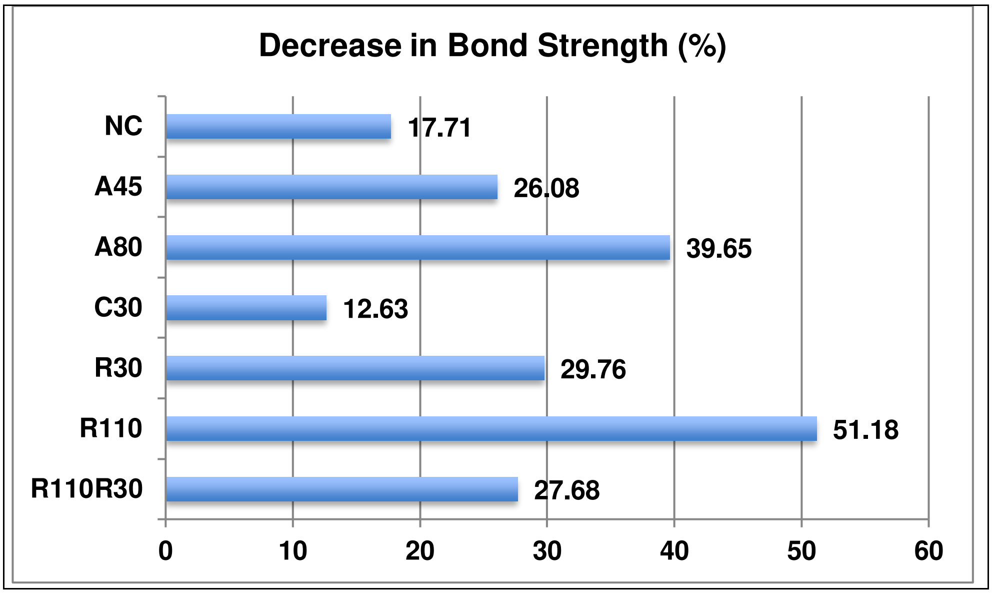
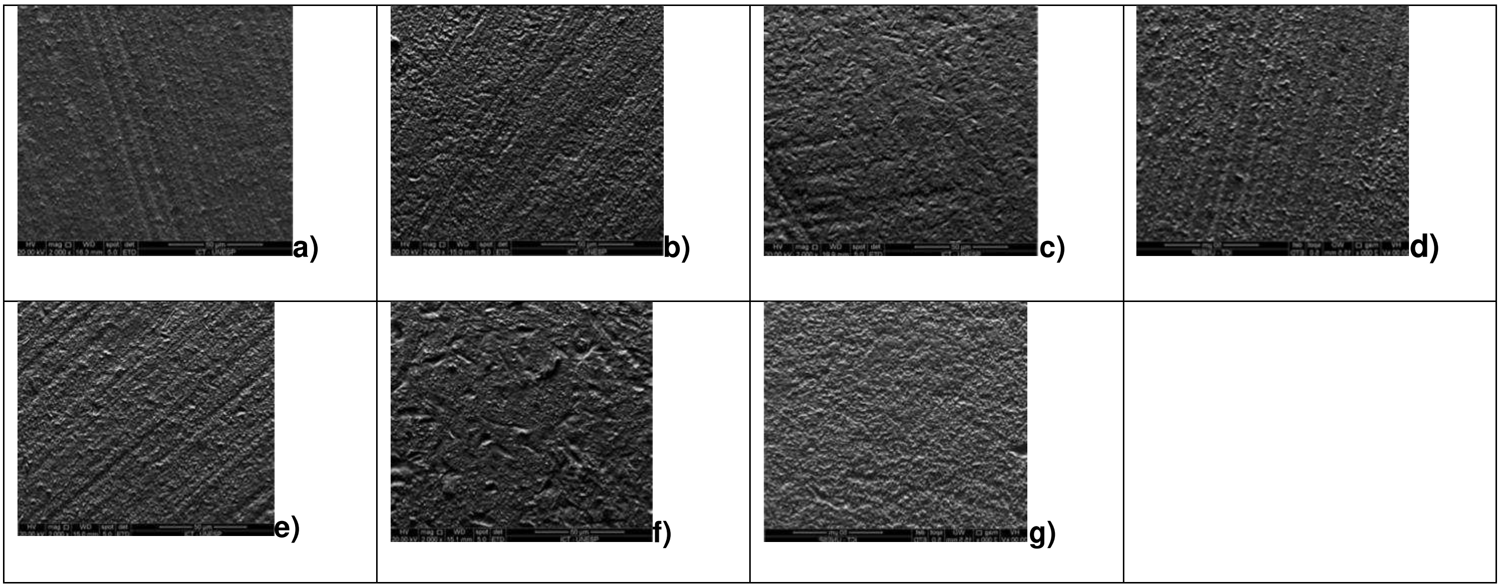


Fig. 1 Bond strength change in percentage between non-aged and aged groups.



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